National Renewable Energy Laboratory

Enzyme Sugar Platform (ESP) Project

Pretreatment

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FY03 Review Meeting

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Pretreatment Outline

- Introduction
  - Technical Barriers
  - Goals
- Equipment and Methods
- Recent Results
- Other Accomplishments
- Recommendations
- Team Members

Pretreatment is Important!

- Key front-end processing step
  - Hydrolyzes hemicellulosic sugar
  - Increases susceptibility of cellulose to enzymatic hydrolysis
  - Required to enable process integration
- Major cost center in overall economics
  - High-yield performance at low cost essential for economic feasibility

Technical Barriers
Pretreatment Specific

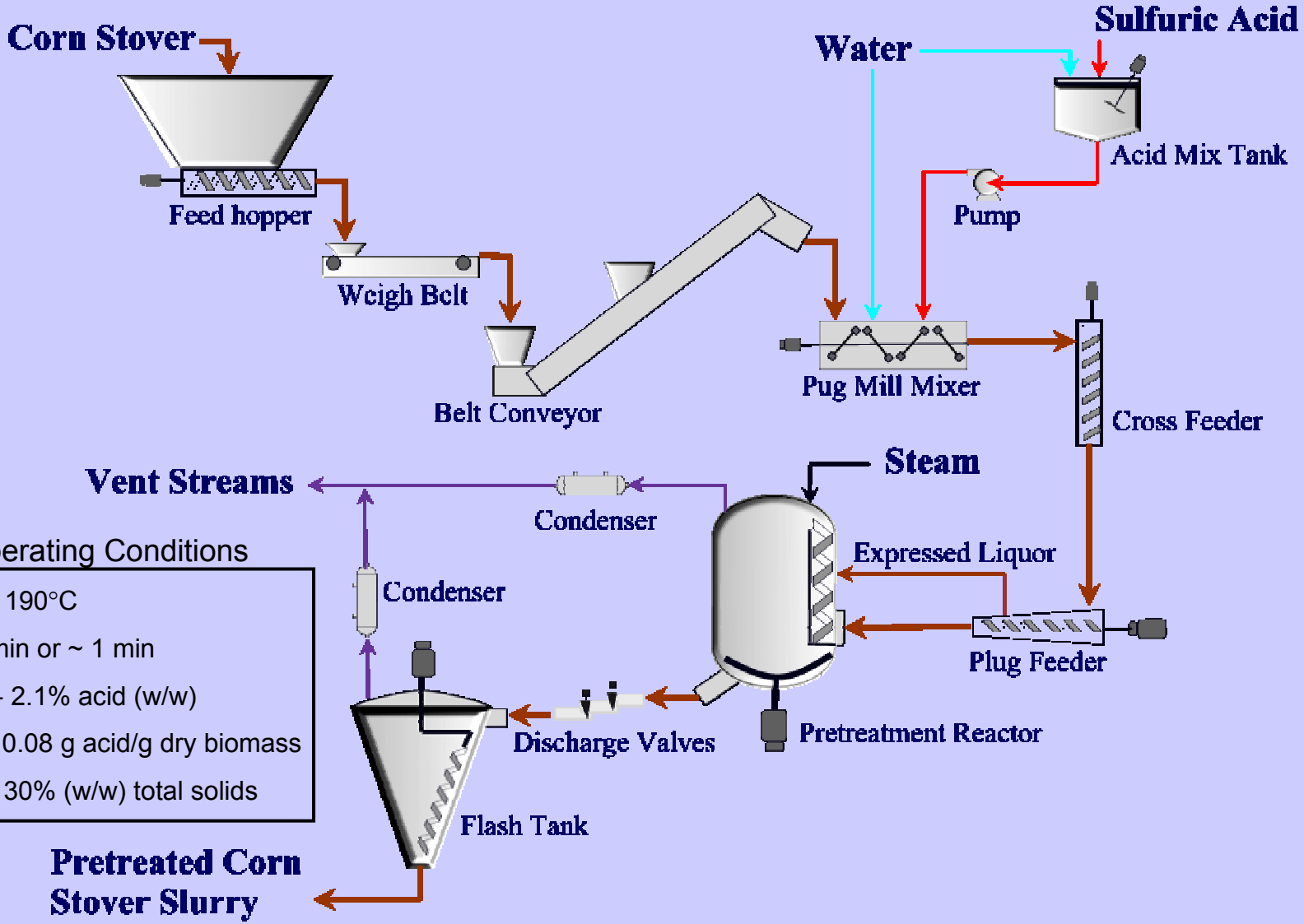
1. Root cause of biomass recalcitrance
2. Role of biomass structure and composition on pretreatment performance
3. Pretreatment process chemistries and reaction kinetics
4. Equipment reliability and materials of construction issues
5. All under realistic high-solids operating conditions

Pretreatment Work Goals

- Supplying process materials
  - Enzyme developers, co-product and combustion studies
- Address/overcome major technical barriers
  - Performance at high-solids concentrations
  - High quality carbon and mass balances (by applying new/improved analytical techniques)
  - Better understand factors affecting cellulose hydrolysis

➢ *Findings and tools are expected to translate to other pretreatment technologies*

Pilot Scale Pretreatment System



Operating Conditions

- 165° - 190°C
- 3-12 min or ~ 1 min
- 0.7% - 2.1% acid (w/w)
- 0.03 - 0.08 g acid/g dry biomass
- 20% - 30% (w/w) total solids

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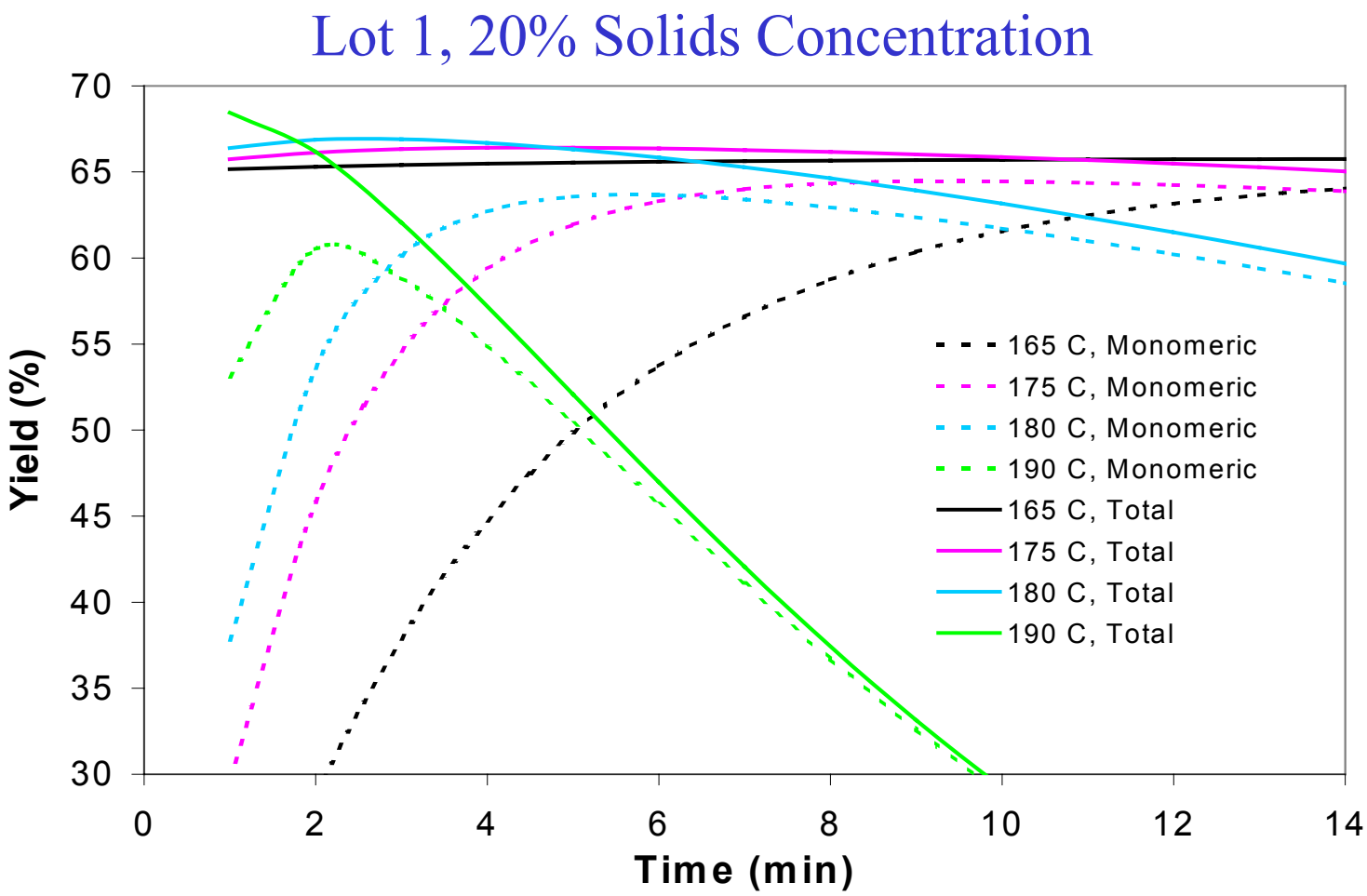
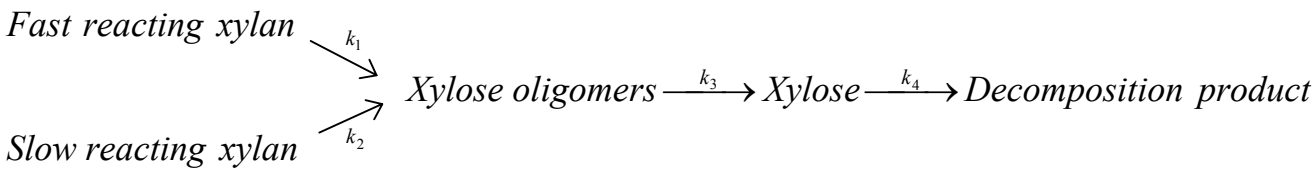
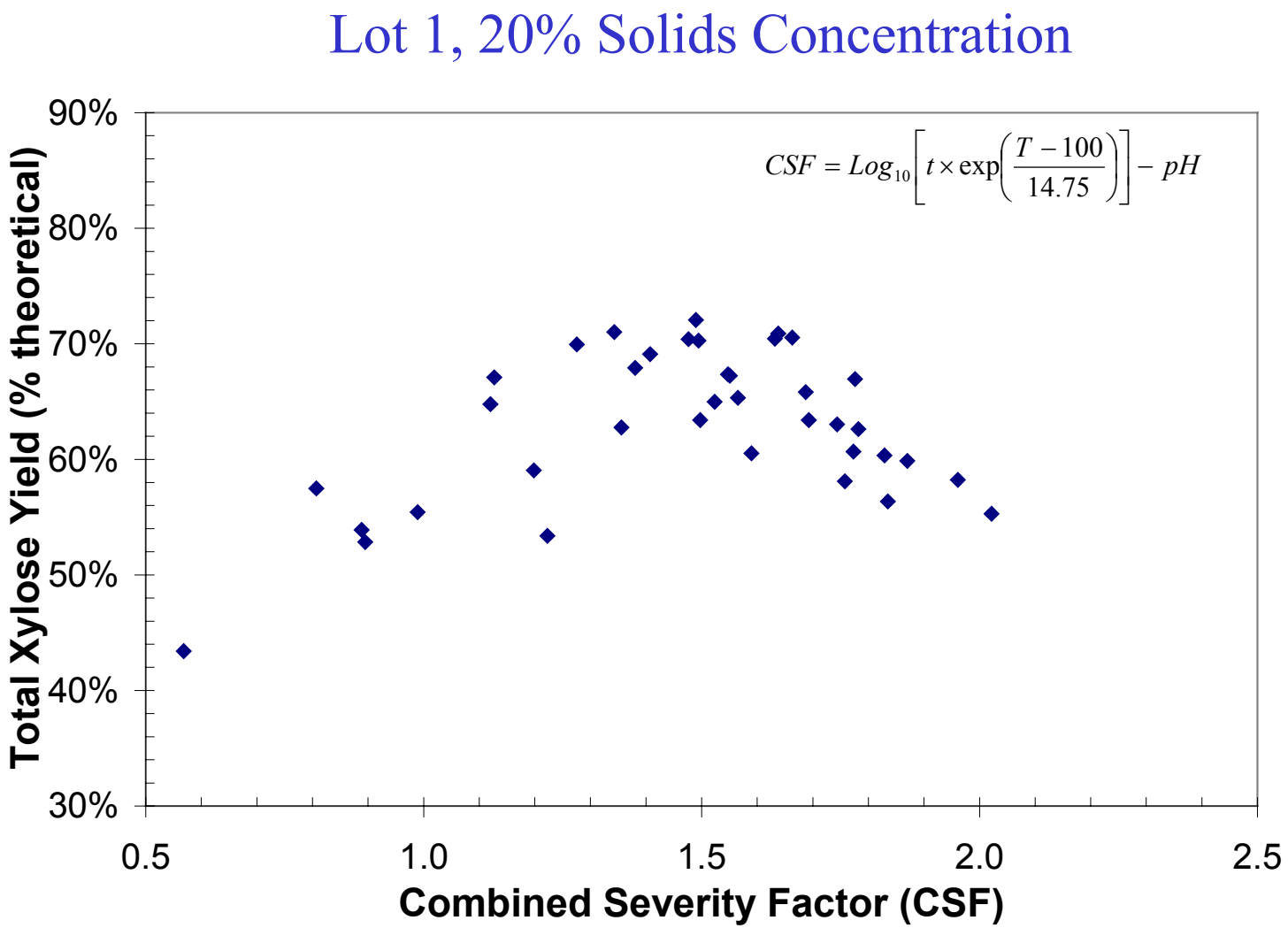
Xylose Yields





Experimental Xylose Yields



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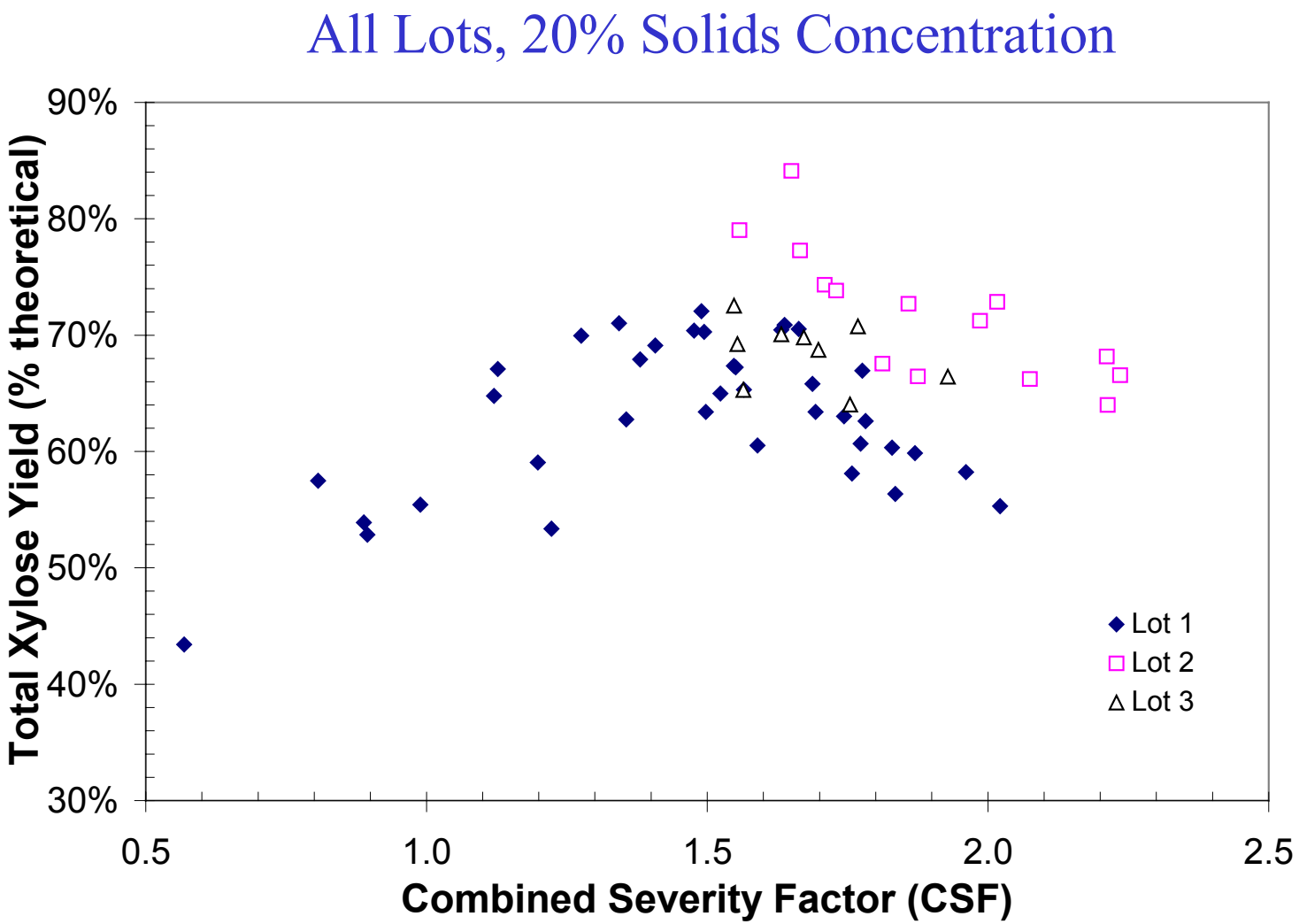
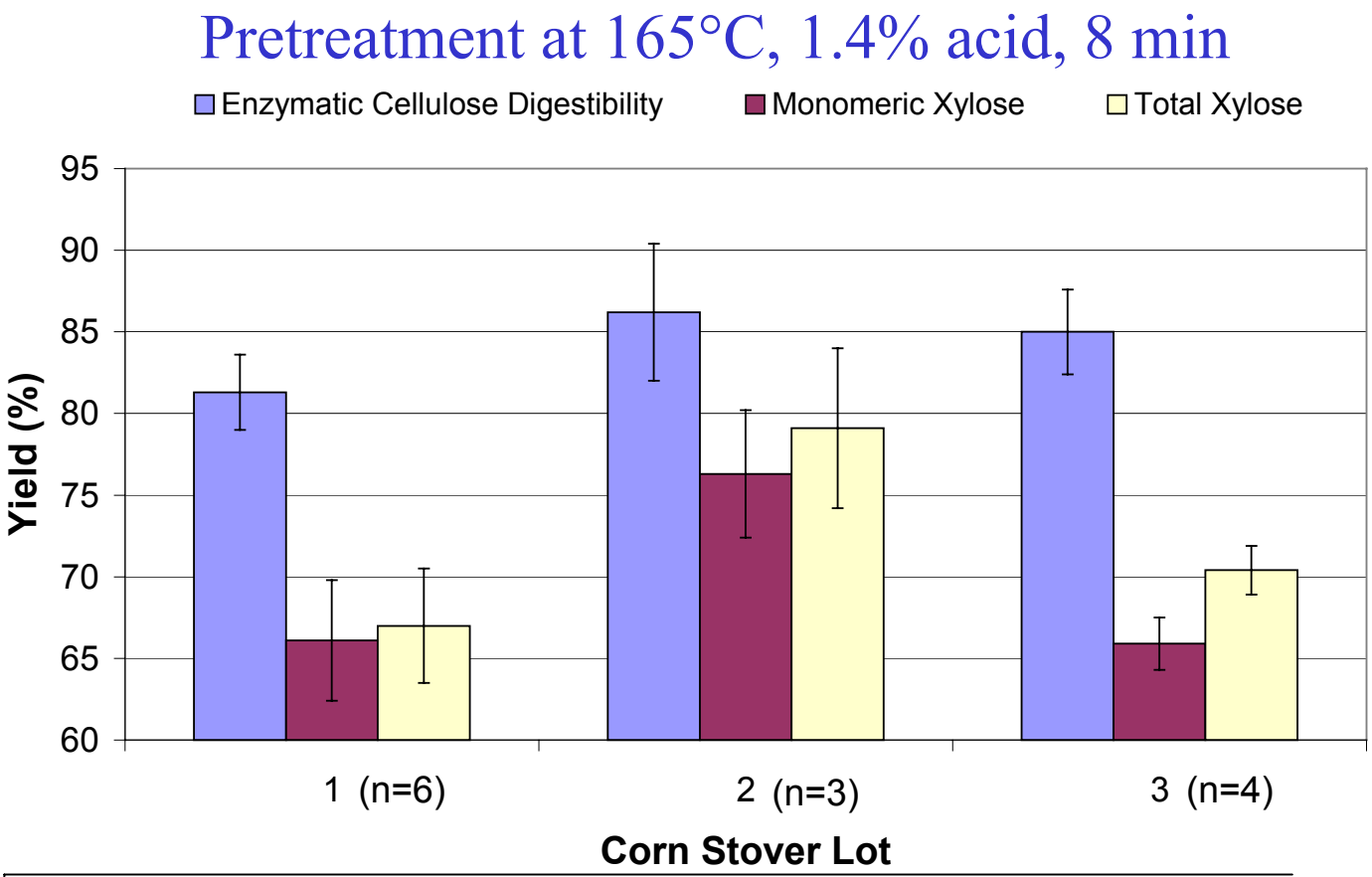


Rigorous Performance Comparison



Cellulose	37.1	38.7	36.6
Xylan	19.9	23.2	21.6
Total	57.0	61.9	58.2

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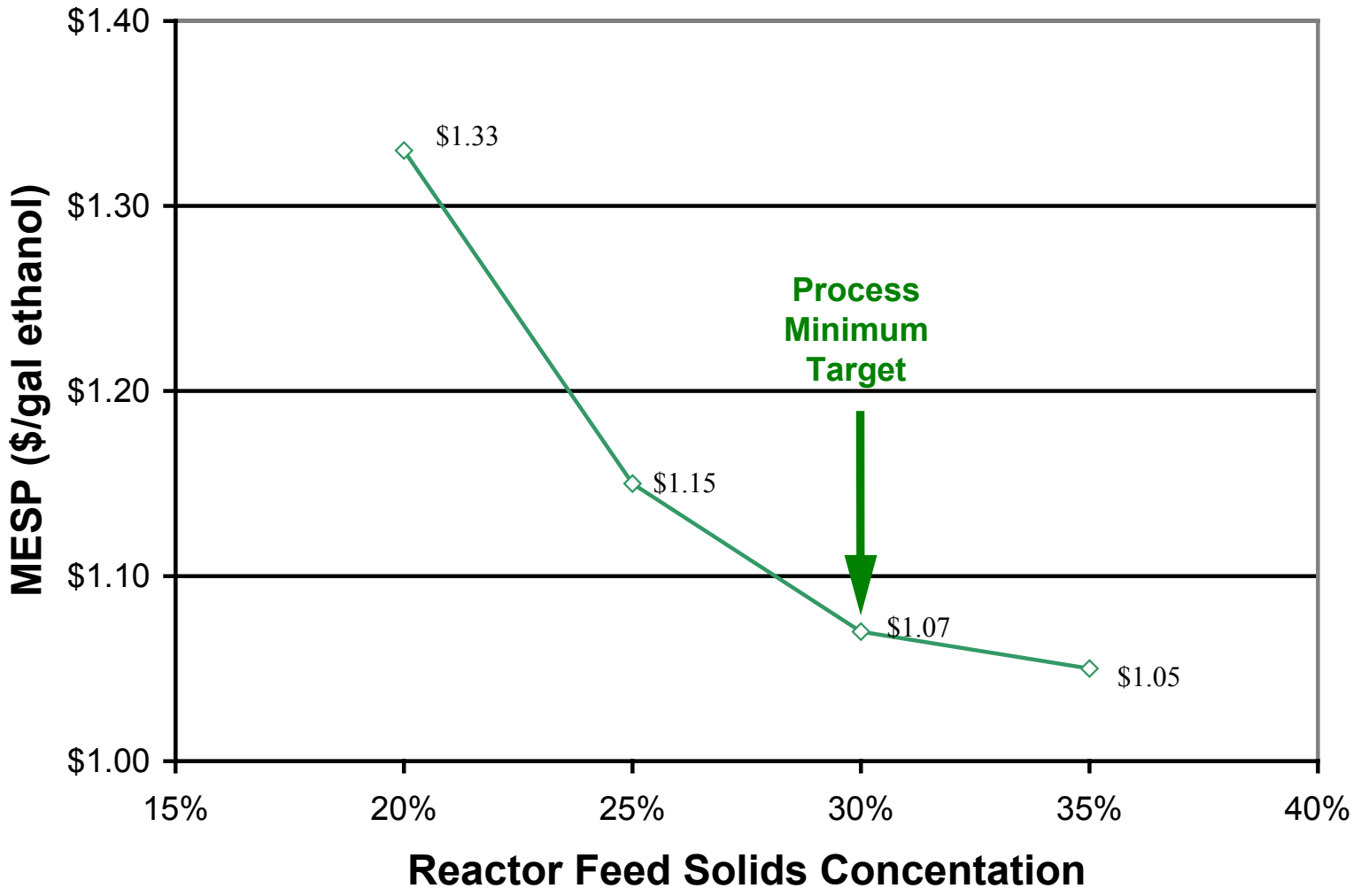
Impact of Pretreatment Solids Concentration

Pretreatment Solids Concentration (wt %)	Total Sugar Concentration (g/L)	Monomeric Xylose Yield (%)	Total Xylose Yield (%)	Xylose Mass Balance Closure (%)	Enzymatic Cellulose Digestibility (%)
20	94	78	85	104	93
30	143	75	78	89	95

Pretreatment at 190°C, 0.048 g acid/g dry biomass, flow-through mode

Recently we successfully operated at 35% solid concentration

Economic Impact of Solids Concentration



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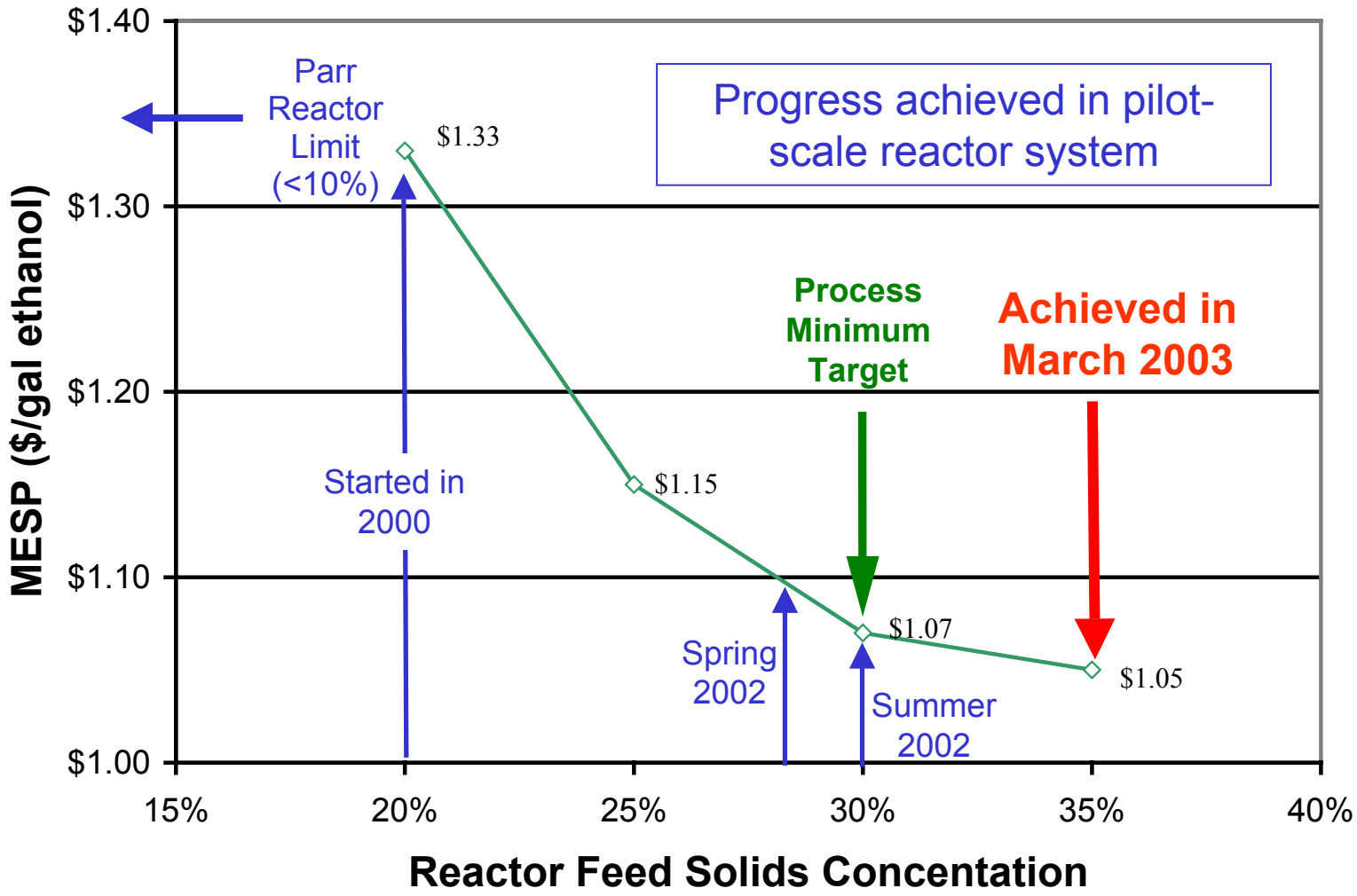
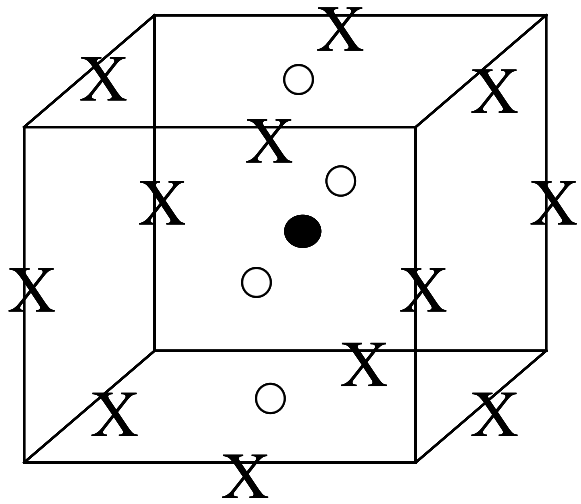
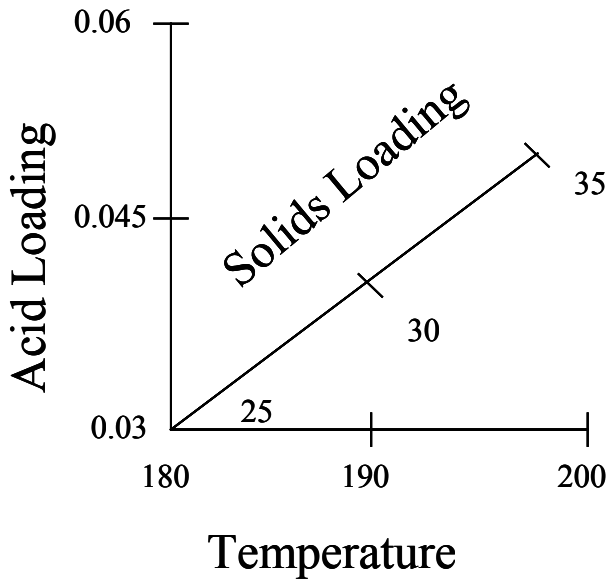


Characterizing High-Solids Performance

Flow-through mode of operation





Measure:

- Component yields/balances
- Enzymatic cellulose conversion
- Characterize structure

Supplying External Stakeholders

2002

Material	Amount Supplied	Number of Contacts Supplied
Raw corn stover	Over 5 tons	4
Pretreated corn stover solids	30 kg (dry)	3
Pretreated corn stover liquor	193 L	5
Enzymatically-digested residue	67 kg (dry)	8

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High Priority Recommendations

- Continue to supply stakeholders
- Characterize high-solids pretreatment
  - Determine component yields/balances
  - Assess potential mass transfer limitations
  - Utilize newly developed assays for uronic acid and soluble protein to improve overall mass balance
    - High quality data necessary for accurate technoeconomic estimates
    - Identify analytical needs to improve mass closures to 100% ± 5%
- Advance understanding of the factors controlling cellulose reactivity to enable predictive modeling
  - Explore enzyme adsorption, accessible pore volume, and other surface characterization tools

Other Recommendation

- Explore effect of corn stover compositional variability on pretreatment performance
  - Improves process modeling and uncertainty assessments
  - Assess impact on process integration

Future New Directions

- Implement new concepts for improving pretreatment performance at the pilot scale
  - Coordinate with Advance Pretreatment Task and input from industry-led projects

Team Members

- Pilot Plant Operation
  - Jody Farmer, Wesley Hjelm, Bob Lyons
- Sample Analysis
  - Bonnie Hames, Bob Lyons, Ray Ruiz, Amie Sluiter,
- Enzymatic Cellulose Conversion Testing
  - Nancy Dowe, Millie Newman